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For and on behalf of RWS Group Ltd

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The 24th day of July 2008

FEDERAL REPUBLIC OF GERMANY



Priority Certificate for the filing of a Patent Application

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Method for improving the durability, dimensional stability and

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Munich, 16 October 2003

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Faust

REHBERG+HÜPPE 16571

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METHOD FOR IMPROVING THE DURABILITY, DIMENSIONAL STABILITY AND SURFACE HARDNESS OF A WOOD BODY

10 The invention relates to a method for improving one or more properties of a wood body, in particular the durability, the dimensional stability and the surface hardness, by impregnating the wood body with an aqueous solution of an impregnating agent and a catalyst and then curing the impregnating agent in the wood body. 15 The invention relates in particular to wood bodies of large dimensions. Wood bodies are understood as meaning a shaped body comprising solid wood. The wood body and the impregnating agent are so to speak to be converted 20 into a composite material in which the positive properties of the natural material wood, in particular the esthetic appearance, are retained but one or more mechanical and biological properties are substantially improved.

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PRIOR ART

The application, "Treatment of timber with water soluble dimethylol resins to improve the dimensional stability and durability", which appeared in Wood Science and Technology 1993, pages 347-355, discloses the treatment of wood with an impregnating agent which consists of solution an aqueous of **DMDHEU** (dimethyloldihydroxyethyleneurea) and a catalyst to improve the shrinkage and swelling properties of wood and the resistance to fungi and insects. Aluminum sulfate, citric acid and metals, individually or in

combination with metals, are used as catalysts. The is DMDHEU used in the aqueous solution concentrations between 5% and 20%. The added amount of catalyst is 20%, based on the DMDHEU. The impregnation takes place in vacuo. At elevated temperature, 5 DMDHEU reacts with itself and with the wood. reaction takes place in the course of one hour in a drying oven at temperatures of 80°C or 100°C. The resin The wood samples thus treated 10 improvement in the shrinkage and swelling properties of up to 75%, in particular at DMDHEU concentrations of 20%. In this way, wood bodies having dimensions of 20 mm \times 20 mm \times 10 mm were investigated. The method described can be used only in the case of small dimensions of the wood bodies because these tend to 15 crack in the case of larger dimensions.

publication by W.D. Ellis, J.L. O'Dell "Wood-The Composites Made with Acrylic Monomers, Isocyanates, and Maleic Anhydride", published of Journal Applied Polymer Science, pages 2493-2505 (1999), discloses the treatment of natural wood with a mixture of acrylates, isocyanate and maleic anhydride in vacuo. The substances used react with themselves but not with the wood. As a result of such an impregnation, the density, hardness and the resistance to water vapor diffusion increase. The water repellency and dimensional stability of the wood are also improved.

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EP 0 891 244 B1 discloses the impregnation of wood bodies comprising solid wood with a biodegradable polymer, a natural resin and/or a fatty acid ester - optionally with the use of vacuum and/or pressure. The impregnation takes place at elevated temperatures. The pores in the wood are at least substantially filled and a shaped body which contains both wood and

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biodegradable polymer forms. A reaction of the polymer with the wood does not take place. With this treatment, the characteristic properties of wood, in particular the water absorption and release, the biodegradability and the mechanical properties are not lost. The thermoplasticity can be increased. Depending on the proportion of polymer introduced, there is an increase in the surface hardness due to the incorporation of the polymer into the wood matrix, so that said timbers are by their own nature also suitable for high-quality floors.

OBJECT OF THE INVENTION

15 It is the object of the invention to provide a method for improving the durability, dimensional stability and surface hardness of a wood body, also having relatively large dimensions, so that various advantageous potential applications result for the wood bodies wherever wood is exposed to moisture and weathering, in particular in case of use as window scantlings, façade boards or steps of staircases.

ACHIEVEMENT

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The object of the invention is achieved, according to the invention by the features of the independent Patent Claim 1.

30 DESCRIPTION OF THE INVENTION

What is concerned here is the improvement of a plurality of properties of wood bodies having relatively large dimensions, for example having a width of 30 to 200 mm and a thickness of 30 to 100 mm. On impregnation of the impregnating agent, surprisingly no cracking occurred, in particular also in the case of

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relatively large dimensions of the wood bodies. Such an impregnation simultaneously improves the durability, dimensional stability and surface hardness of the wood body.

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The use of the impregnating agent in pure or in modified form is significant for the invention. The wood body is completely impregnated throughout during the impregnation. The impregnation can be carried out under the action of vacuum with subsequent action of pressure. This is expedient particularly in combination with high proportions by weight of the impregnating agent.

- 15 DMDHEU, as a substance of group A, is known as an impregnating agent for wood samples:
 - 1,3-bis(hydroxymethyl)-4,5-dihydroxyimidazolidin-2-one (DMDHEU).

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The following substances of group B have not been known to date as impregnating agents for wood:

- Urea-glyoxal adducts and derivatives thereof:
 derivatives of 1,3-bis(hydroxymethyl)-4,5-dihydroxyimidazolidin-2-one (mDMDHEU),
 1,3-dimethyl-4,5-dihydroxyimidazolidin-2-one (DHDMI).

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The following substances of group ${\bf C}$ can be used as catalysts:

5 1. Salts:

Chlorides, e.g. MgCl₂; ZnCl; LiCl,

Ammonium salts, e.g. ammonium chloride; ammonium sulfate; ammonium oxalate,

Phosphates, e.g. diammonium phosphate,

Nitrates, e.g. $ZnNO_3$,

Borates, e.g. sodium tetrafluoroborate.

2. Acids:

Maleic acid,

15 Formic acid,
Hydrochloric acid,
Sulfuric acid.

All substances of groups A and/or B can be used in combination with one another. Said impregnating agent may also be a product which contains one or more of these substances. Combinations of the substances from group C can also be used as the catalyst.

It is particularly important to maintain moist conditions during the curing of the impregnating agent so that drying during this reaction is thus avoided. The impregnating agent introduced into the wood is thus caused to react with itself and the wood under moist conditions and with avoidance of drying. The use of a temperature in the range from 80°C to 100°C is possible if only a drying effect is avoided.

In particular, magnesium chloride can be used as the 35 catalyst.

Further details of the method form the subject matter

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of subclaims.

The invention also relates to the use of an impregnating agent containing one or more of the substances of group B for improving one or more properties of a wood body. This applies to wood bodies of smaller and larger dimensions.

The use of an impregnating agent containing one or more substances of the groups A and/or B in a concentration of 1% by weight to 50% by weight in the aqueous solution leads to an improvement in a plurality of properties of a wood body. It is also possible to use all measures which were described in relation to the method.

DESCRIPTION OF PREFERRED EMBODIMENTS

Embodiment 1

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It is intended to produce window scantlings from *Pinus radiata*, i.e. profiled shaped bodies which can be used in the production of window frames. mDMDHEU of a commercially available aqueous solution is diluted to 50% by weight with water and mixed with 1% of MgCl₂. The wood bodies dried to about 12% wood moisture are introduced into an impregnating unit. The impregnating unit is exposed to a vacuum of 50 mbar absolute for one hour. This is followed by flooding of the impregnating unit with the impregnating solution. The vacuum of 50 mbar absolute is kept constant. A pressure of 12 bar is then applied for 2 hours. The pressure phase is terminated and the residual liquid is removed.

35 The wood bodies are then stored in a drying chamber controllable via temperature and atmospheric humidity and fixed so that warping is counteracted. The chamber

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is brought to 80°C and a relative humidity of about 95%. These moist conditions are maintained until a temperature of about 80°C was reached for 72 hours in the interior of the wood bodies.

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There follows a drying process at a temperature of about 50°C and for a duration of 14 days.

Embodiment 2

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Here, round pine posts are to be treated so that palisades can be erected therewith. DMDHEU in a commercially available aqueous solution is diluted to about 10% by weight with water and mixed with 10% of ZnNO₃, based on the amount of DMDHEU. The round wood bodies dried to about 20% wood moisture and having about the same dimensions are introduced into an impregnating unit. This impregnating unit is flooded with the impregnating solution and for a pressure of 12 bar is applied for 2 hours. The pressure phase is terminated and the residual liquid is removed.

The wood bodies are then stored in a drying chamber controllable via temperature and atmospheric humidity and fixed so that warping is impossible. The drying chamber is brought to 99°C and a relative humidity of about 80%. The conditions are maintained until a temperature of at least 98°C was reached for 24 hours in the interior of the wood bodies. Thereafter, the wood bodies can be dried on a thoroughly ventilated wood stack in the open air.

Embodiment 3

35 It is intended to treat poplar boards in order to produce decks for outdoor applications.

Tetramethylolacetylenediurea of a commercially available solution is diluted to 10% by weight with and mixed with 1% of sodium tetrafluoroborate. The boards dried to about 12% by weight wood moisture are introduced into an impregnating unit. This is flooded with the impregnating solution and is exposed to a vacuum of 50 mbar absolute for 1 hour. After the end of the vacuum phase, the residual moisture is removed.

10 The shaped bodies are heated to about 80°C in a saturated water vapor atmosphere. This can be effected, for example, by packing the wood bodies in film which remains stable at this temperature. The duration of the action of heat is dependent on the type of wood and the dimensions of the wood bodies. In the case of 6 to 7 cm thick wood bodies, the reaction time is about 50 hours.

The wood can be fixed in stacks so that warping is made impossible. A drying process takes place for 14 days under standard conditions of temperature and humidity, inside or outside.

Embodiment 4

25 It is intended here to produce staircase steps comprising solid wood, for example beech. The steps may have dimensions of 1000 mm \times 400 mm \times 80 mm. Particular value is placed on an increased surface hardness of the staircase steps.

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DMU is diluted to an aqueous solution to 1% by weight with H_2O and mixed with 0.5% by weight of $ZnNO_3$. The staircase steps dried to about 12% wood moisture and having about the same dimensions are introduced into an impregnating unit which is flooded with impregnating solution. In the impregnating unit, a vacuum of 40 mbar absolute is established for 1 hour. The impregnating

unit is then brought to a pressure of 8 bar for 2 hours. After the end of the pressure phase, a vacuum of 200 mbar is applied for 10 min. The vacuum phase is then terminated and the residual moisture is removed.

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The staircase steps are heated in a water-saturated atmosphere to about 95°C. For this purpose, the staircase steps are first packed in film which remains stable at a temperature of 95°C. The duration of the action of heat is dependent on the type of wood and the dimensions of the steps. In the case of steps having a thickness of 80 mm, the reaction time is about 70 hours.

15 After the reaction, the wood is fixed in stacks so that warping is made impossible. There follows a drying process at a temperature of about 40°C and for a duration of 20 days. A conventional drying chamber can be used for this purpose.

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PATENT CLAIMS

- Method for improving the durability, dimensional 1. stability and surface hardness of a wood body by 5 impregnating the wood body with an solution of an impregnating agent comprising the substance of group A and/or one or more substances of group B and one or more substances of group C as a catalyst, the impregnating agent comprising 10 the substance of group A and/or the substance or οf group В being substances used concentration of 1% by weight to 50% by weight in the aqueous solution, and the impregnating agent then being caused to react with itself and with 15 the wood under moist conditions with avoidance of drying.
- 2. Method according to Claim 1, characterized in that the impregnating agent introduced into the wood is caused to react with itself and with the wood under the moist conditions at a temperature in the range from 80°C to 100°C.
- 3. Method according to at least one of Claims 1 and 25 2, characterized in that magnesium chloride is used as the catalyst.
- 4. Method according to at least one of Claims 1 to 3, characterized in that a vacuum of about 50 mbar is used for about one hour.
 - 5. Method according to at least one of Claims 1 to 4, characterized in that a pressure of about 12 bar is used for about 2 hours.
 - 6. Method according to at least one of Claims 1 to 5, characterized in that the reaction of the

impregnating agent takes place at below about 100°C for a duration of about 48 h.

- 7. Method according to at least one of Claims 1 to 6,

 5 characterized in that the catalyst is used with a proportion of up to about 10% by weight based on the amount of chemicals in the aqueous solution.
- 8. Method according to at least one of Claims 1 to 7,

 characterized in that, after the impregnation, the wood body is fixed so that a change in the shape of the wood body during the curing of the impregnating agent is counteracted.
- 9. Method according to at least one of Claims 1 to 9, characterized in that the reaction of the impregnating agent with the wood body is carried out at a relative humidity of above 80%.
- 20 10. Use of an impregnating agent containing one or more of the substances of group B for improving a property of a wood body.

ABSTRACT

A method for improving the durability, dimensional stability and surface hardness of a wood body by impregnating the wood body with an aqueous solution of an impregnating agent comprising the substance of group A and/or one or more substances of group B and one or more substances of group C as a catalyst is described. The impregnating agent comprising the substance of group A and/or the substance or substances of group B is used in a concentration of 1% by weight to 50% by weight in a aqueous solution. The impregnating agent is then caused to react with itself and with the wood under moist conditions with avoidance of drying.